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Mineralogical Identification on Polluted Soils using XRD Method

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Abstract

Soil samples were collected from an industrial area at Karaikal, South India. Mineralogical identification was carried out on the soil samples using powder X-Ray Diffraction (XRD) method. The XRD results indicated the presence of various minerals, namely quartz, kaolinite, hematite, aragonite, illite and calcite. XRD Method is non-destructive and can be used in the identification of mineralogical composition. Results were discussed and it was arrived that the method is relatively quicker and more reliable in mineral analysis.

Keywords: *Industrial Pollution; Mineral analysis; Powder X-ray diffraction; Soil.*

1. INTRODUCTION

Pollution is the introduction of contaminants into a natural environment that causes instability, disorder, harm or discomfort to the ecosystem i.e. physical systems or living- organisms. Pollution denotes the presence of impurities like dirt, silt, organic matter, minerals, acidity, alkalinity and objectionable colour, odour and taste. Although pollution is not necessarily a health hazard always, it is often accompanied by contamination which is a health hazard. Pollution of water, air and soil environment due to industrial and other waste is one of the problems faced by the developed as well as developing countries (Prasanthan and Nayar, 2000).

Sediments are principal carriers of the trace elements in the hydrosphere. Sediment particles are made up of materials derived from rock, soil, biological and anthropogenic inputs. The basic structural unit of inorganic sediment is silicate and aluminosilicate. Major components of sediments include clay, quartz, feldspars, various silicate minerals, gibbsite and calcium carbonate. The properties of sediment depend on its mineral composition, percentage of organic matter, sorption capacity for pollutants, porosity and particle size distribution. Properties of the sediments decide the concentration of the pollutant in the sediment. The mineral analysis is the prominent area of there search on environmental pollution. The techniques such as thin section analysis, differential thermal analysis, X-ray diffraction studies, Mossbauer studies, magnetic methods, ultra-violet absorption studies and Infrared spectroscopic analysis are used for mineral analysis (Ravisankar *et al*. 2010). XRD method is the best one for mineral analysis as it is rapid, cheap and is not time consuming and is non-destructive. The Powder X-ray diffraction pattern contains more information about mineralogy (Ramasamy *et al*. 2006). It is used by mineralogists and sedimentary petrologists in the aspect of mineralogical application. The principal

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constituents of most of the sediments are quartz, feldspar and clay minerals. One of the most important and value added applications of the XRD study is the identification of the minerals in the sediment samples. The most widely occurring clay minerals in soil or sediments are kaolinite which is the simplest of all the clay minerals (Chapman *et al.* 2001). Quartz is usually an important mineral because of the formation of soil from highly silica material. XRD technique is used to distinguish the different type of clay minerals and to derive information concerning their structure, composition and structural changes upon chemical modification (Madejova, 2003). Pollutant soil varies widely in composition and physical characteristics as a function of water depth, distance from land, variation in soil source and the physical, chemical and biological characteristics of the environment. A large number of researchers determined semi-quantitative clay mineral composition on the basis of area under X-ray diffraction peak duly corrected by appropriate factors accounting for variation of scattering due to variation of angle (Nayak and Singh, 2007).

In the present study an attempt has been made to investigate the qualitative mineral analysis of polluted soil samples. XRD method is ananalytical tool that presents a lot of advantages and it is accurate, inexpensive and reliable.

2. MATERIALS & METHODS

In the present study, 36 soil samples were collected from four canals flowing around a chemical industry located near Karaikal Port at Karaikal, Pondicherry State, India using standard procedures. Each location is separated by a distance of 100 m approximately. All the samples were collected during the summer season of 2012. In each site, three samples were collected, one at the surface level, second at 15 cm depth from the surface and the third at 30 cm depth. 6 samples were selected for the present study. Those 36 samples were dried at room temperature in open air for two days and stored in black polythene bags. The soil samples were well powdered by using an agate mortar and then oven dried at 60 °C for two hours to remove the moisture content. The XRD analysis was performed with Model XD-D1 Shimadzu Diffractometer with CuKα radiation of λ= 1.54Å, operating at 30 kV and 30 mA, available at Solid State Structural and Chemistry Unit, Indian Institute of Science, Bangaluru, India.

A flat specimen is mounted on a turn table around which moves a detector. The sample changes the angle of incident beam as it rotates. Whenever the Bragg condition for X-ray diffraction is fulfilled, x-rays are diffracted to the detector. A large number of sets of parallel planes are possible in each crystal. Each set of planes has its own value of θ for which the incident X-ray beam will be diffracted and θ is related to the interplanar spacing “d”. Reinforcement will only take place when the X-rays diffracted by parallel planes of atoms are in phase with one another; that is when their path difference is an integral number of times the wavelength. Bragg’s law represents this condition.

$$2d \sin \theta = n\lambda$$

where, λ is the wavelength, d - interplanar spacing, θ–angle of incidence (and diffraction) of the X-rays and ‘n’ is the order. The ‘d’ values of each pattern are compared with the standard data published by Joint Committee on Powder Diffraction Standards JCPDS.

3. RESULTS & DISCUSSION

XRD pattern of select 6 soil samples (at the surface layer, at 15 cm depth and at 30 cm depth) are quality analyzed. Samples were numbered as S19, S20, S21 and S28, S29 and S30. This study is used to bring out the mineralogical composition and to analyze the crystalline nature of the minerals. Selected representative XRD patterns of soil samples in different locations at two canals (Canals 3 & 4) are shown in Fig. 1 & Fig. 2. The observed data from the entire XRD pattern are given in the Tables 1-6 along with their

corresponding mineral names. The minerals such as quartz, kaolinite, hematite, calcite, aragonite, feldspar, and illite are identified. For all the samples, '2θ' values and intensities of the experimental data were compared with the standard data collected from the Joint Committee on Powder Diffraction Standards.

XRD is used to determine the mineralogical composition of the soil samples as well as qualitative and quantitative phase analysis of multiphase mixtures (Nayak and Singh, 2007). The presence of minerals in clay is identified by comparing '2θ' values. The possible minerals with their '2θ' values present in the adsorbents are given in Tables 1 to 6. The XRD pattern indicates the presence of quartz, kaolinite, hematite, calcite, aragonite, feldspar and illite. Further, the presence of the above minerals in the soil samples is confirmed by FT-IR study (Oumabady *et al.* 2013).

In the third canal, the three soil samples (S_{19}-S_{21}) were found to have minerals such as quartz, kaolinite, illite, hematite, aragonite and feldspar with quartz and kaolinite being the major constituents. The other minerals namely, hematite, feldspar, aragonite and calcite are present as minor constituents. The XRD patterns of the samples under investigation are analyzed; the '2θ' values of samples are compared with standard values from JCPDS data. The overlaps of XRD patterns are given in Fig.1. Experimental '2θ' values, relative intensities and corresponding standard

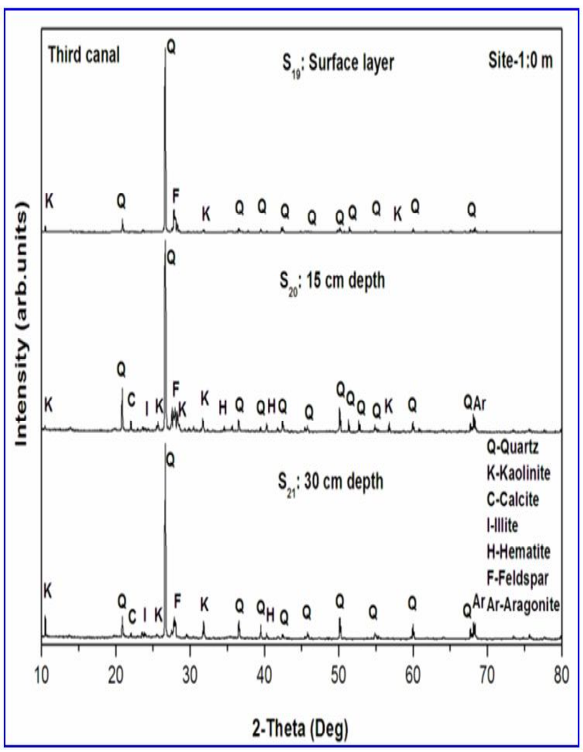

Fig. 1: XRD pattern of soil in third canal from site 1 samples (S19-S21)

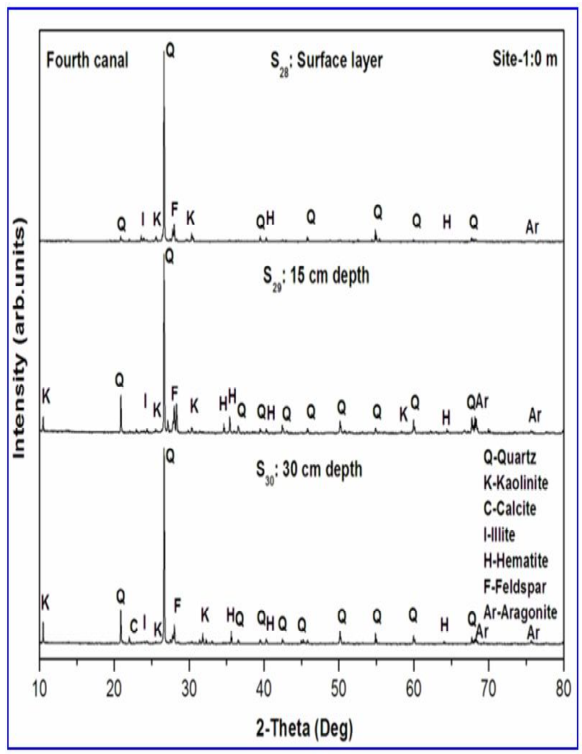

Fig. 2: XRD pattern of soil in fourth canal from site 1 samples (S28-S30)

Table 1. Comparison between observed and standard values of ‘2θ’ and I/I_0 of soil sample (S19)

Observed 2θ	Standard value			Mineral	JCPDS
	2θ	I/I_0	hkl		
10.482	10.706	60	021	kaolinite	02-0105
20.840	20.862	219	100	Quartz	85-1053
26.639	26.644	999	011	Quartz	85-1053
27.961	27.702	759	002	Feldspar	70-2121
31.942	31.901	40	154	kaolinite	02-0105
36.528	36.551	67	110	Quartz	85-1053
39.655	39.504	65	102	Quartz	85-0594
42.424	42.459	48	200	Quartz	85-1053
46.294	45.898	29	201	Quartz	85-1053
50.151	50.245	10	112	Quartz	85-0865
51.473	51.515	11	003	Quartz	83-2473
54.972	54.923	27	002	Quartz	85-0794
57.630	57.383	11	242	kaolinite	89-6538
60.041	60.009	74	121	Quartz	85-0794
67.988	67.973	2	104	aragonite	67.973

Table 2. Comparison between observed and standard values of ‘2θ’ and I/I_0 of soil sample (S20)

Observed 2θ	Standard value			Mineral	JCPDS
	2θ	I/I_0	hkl		
10.715	9.89	1	10	kaolinite	89-5695
21.132	20.862	219	100	quartz	85-1053
22.024	22.301	68	201	calcite	87-1863
24.187	24.318	40	112	illite	29-1496
26.699	26.644	999	11	quartz	85-1053
27.837	27.702	759	002	feldspar	70-2121
28.223	28.251	35	112	kaolinite	83-0971
36.712	36.551	67	110	quartz	85-1053
39.635	39.475	65	102	quartz	85-1053
40.475	40.321	202	113	hematite	88-2359
42.558	42.666	51	200	quartz	85-1053
46.010	45.898	29	201	quartz	85-1053
50.457	50.245	10	112	quartz	85-0865
55.059	55.327	15	13	quartz	85-1054
58.822	58.675	3	241	kaolinite	83-0971
60.190	60.17	70	211	quartz	85-1780
63.114	63.441	15	113	quartz	85-1780
68.400	68.685	27	242	aragonite	76-0606

Table 3. Comparison between observed and standard values of '2θ' and I/I_0 of soil sample (S21)

Observed 2 θ	Standard value			Mineral	JCPDS
	2 θ	I/I_0	hkl		
10.592	10.706	60	021	kaolinite	02-0105
20.964	20.960	228	100	quartz	85.1780
22.024	22.301	68	201	calcite	87-1863
24.187	24.318	40	112	illite	29-1496
25.909	25.994	19	111	kaolinite	89-6538
26.639	26.644	999	11	quartz	85-1053
27.961	27.702	759	002	feldspar	70-2121
31.818	31.901	40	154	kaolinite	02-0105
36.528	36.551	67	110	quartz	85-1053
39.531	39.504	65	102	quartz	85-0594
40.491	40.321	202	113	hematite	88-2359
42.672	40.832	191	113	hematite	89-2810
45.927	45.898	29	201	quartz	85-1053
50.275	50.245	10	112	quartz	85-0865
54.972	54.923	27	002	quartz	85-0794
60.151	60.170	70	211	quartz	85-1780
67.754	67.766	42	212	quartz	85-0930
68.360	68.497	40	231	aragonite	03-1067

Table 4. Comparison between observed and standard values of '2θ' and I/I_0 of soil sample (S28)

Observed 2 θ	Standard value			Mineral	JCPDS
	2θ	I/I_0	hkl		
20.840	20.862	219	100	quartz	85-1053
24.187	24.318	40	112	illite	29-1496
25.426	25.280	40	133	kaolinite	02-0105
26.749	26.696	999	101	quartz	85-0865
28.085	28.251	35	112	kaolinite	83-0971
30.371	30.374	57	222	feldspar	84-0710
39.531	39.504	65	102	quartz	85-0594
40.495	40.321	202	113	hematite	88-2359
45.928	45.917	60	201	quartz	85-0865
55.096	55.327	15	13	quartz	85-1054
60.275	60.170	70	211	quartz	85-1780
66.022	66.092	3	125	hematite	85-0599
67.754	67.766	42	212	quartz	85-0930
75.716	75.706	69	243	aragonite	71-2392

Table 5. Comparison between observed and standard values of ‘2θ’ and I/I_0 of soil sample (S29)

Observed 2 θ	Standard value			Mineral	JCPDS
	2θ	I/I_0	hkl		
12.887	12.355	999	1	kaolinite	89-6538
20.852	20.862	219	100	quartz	85-1053
24.055	24.318	40	112	illite	29-1496
26.699	26.644	999	110	quartz	85-1053
28.098	27.702	759	002	feldspar	70-2121
29.866	29.534	44	112	kaolinite	89-5695
36.712	36.551	67	110	quartz	85-1053
39.76	39.475	65	102	quartz	85-1053
40.475	40.832	191	113	hematite	89-2810
42.683	42.666	51	200	quartz	85-1053
45.886	45.898	29	201	quartz	85-1053
50.333	50.245	10	112	quartz	85-0865
55.134	55.327	15	013	quartz	85-1054
60.191	42.666	51	200	quartz	85-1053
68.400	68.685	27	242	aragonite	76-0606
75.775	75.706	69	243	aragonite	71-2392

Table 6. Comparison between observed andstandard values of ‘2θ’ and I/I_0 of soil sample (S30)

Observed 2 θ	Standard value			Mineral	JCPDS
	2 θ	I/I_0	hkl		
10.592	10.706	60	021	kaolinite	02-0105
20.964	20.960	228	100	quartz	85.1780
22.245	22.301	68	201	calcite	87-1863
24.228	24.318	40	112	illite	29-1496
25.674	25.765	20	151	kaolinite	02-0105
26.749	26.696	999	101	quartz	85-0865
27.961	27.702	759	002	feldspar	70-2121
31.941	31.901	40	154	kaolinite	02-0105
35.674	35.682	716	110	hematite	79-0007
36.639	36.728	65	110	quartz	85-1780
39.531	39.504	65	102	quartz	85-0594
40.385	40.321	202	113	hematite	88-2359
42.672	42.666	51	200	quartz	85-1053
45.206	45.328	40	048	hematite	02-0919
50.275	50.245	10	112	quartz	85-0865
54.845	54.872	30	022	quartz	85-0798
60.041	60.009	74	121	quartz	85-0794
64.225	64.286	229	211	hematite	85-0599
67.875	67.766	42	212	quartz	85-0930
68.363	68.497	40	231	aragonite	03-1067
75.826	75.706	69	243	aragonite	71-2392

values are given in Tables 1-3. The observed XRD patterns of results indicate quartz and kaolinite as the major constituents and other minerals as the minor constituents and are known to crystallize in hexagonal and anorthic system. These results are in good agreement with FT-IR studies (Oumabady *et al.* 2013).

In the fourth canal, the three soil samples (S_{28}-S_{30}) were found to have minerals such as quartz, kaolinite, illite, hematite, aragonite and feldspar with quartz and kaolinite as the major constituents. The other minerals namely, hematite, feldspar, aragonite and calcite are present as minor constituents. The XRD patterns of third canal samples are almost identical to those of fourth canal samples. The XRD patterns of the samples under investigation are analyzed; the '2θ' values of samples are compared with standard values from JCPDS data. The overlaps of XRD patterns are given in Fig. 2. Experimental '2θ' values, relative intensities and corresponding standard values are given in Tables 4-6. The observed XRD patterns of results indicate quartz and kaolinite as the major constituents and other minerals as the minor constituents and they are known to crystallize in hexagonal and anorthic system. These results are in good agreement with FT-IR studies (Oumabady *et al.* 2013).

4. CONCLUSION

The XRD results indicated the presence of various minerals, namely quartz, kaolinite, hematite, aragonite, illite and calcite. XRD method is non-destructive and can be used in the identification of mineralogical composition.

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